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PHYSICOCHEMICAL CHARACTERIZATION AND THERMAL STABILITY OF SULFUR CELLULOSE COMPOSITES OBTAINED BY INVERSE VULCANIZATION

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Abstract. The article presents the results of a comprehensive physicochemical study of sulfur-cellulose composites (SCC) synthesized by inverse vulcanization from waste elemental sulfur (S_8) and activated cotton husk. Chemical interaction of the components was proven by elemental analysis, FTIR spectroscopy, X-ray diffraction (XRD), scanning electron microscopy with energy-dispersive X-ray analysis (SEM/EDX), thermogravimetry (TGA) and differential scanning calorimetry (DSC). The introduction of sulfur groups leads to the disappearance of crystalline sulfur reflections on diffractograms, the appearance of characteristic S–S (470 cm^{-1}) and C–S (420 cm^{-1}) bands in IR spectra, and homogeneous distribution of sulfur and carbon at the microscale. The synthesized composite (SCC-2, S:Org = 55:45) exhibits high thermal stability: the onset decomposition temperature in an inert atmosphere is 272 °C, in an oxidizing atmosphere – 265 °C, which is 50 °C higher than that of a mechanical mixture. The obtained results substantiate the suitability of SCC as a thermostable additive for drilling fluids operating in HTHP conditions up to 180 °C.

Keywords: sulfur-cellulose composite, inverse vulcanization, FTIR spectroscopy, X-ray diffraction, thermogravimetry, thermal stability, polysulfide bonds, drilling fluid.

Annotatsiya. Maqolada invers vulkanizatsiya usuli yordamida chiqindi elementar oltingugurt (S_8) va faollashtirilgan paxta qobig‘i asosida sintez qilingan oltingugurt-sellyuloza kompozitlarining (OSK) kompleks fizik-kimyoviy tadqiqoti natijalari keltirilgan. Element tahlili, Fure IK-spektroskopiyasi, rentgenofazaviy tahlil (XRD), energiyadispersion rentgen tahlili bilan skanerlovchi elektron mikroskopiya (SEM/EDX), termogravimetriya (TGA) va differensial skanerlovchi kalorimetriya (DSK) usullari bilan komponentlarning kimyoviy o‘zaro ta’siri isbotlandi. Oltingugurt guruhlarining kiritilishi oltingugurtning kristall reflekslarining difraktogrammalarda yo‘qolishiga, IK-spektrlarda S–S (470 sm^{-1}) va C–S (420 sm^{-1}) xususiyatli polosalarining paydo bo‘lishiga hamda oltingugurt va uglerodning mikro darajada bir tekis taqsimlanishiga olib keladi. Sintez qilingan kompozit (OSK-2, S:Org = 55:45) yuqori termik barqarorlikka ega: inert atmosferada parchalanish boshlanish harorati 272 °C, oksidlovchi muhitda esa 265 °C ni tashkil etadi, bu mexanik aralashmaga nisbatan 50 °C yuqori. Olingan natijalar OSK ni 180 °C gacha bo‘lgan HTHP sharoitlarida ishlaydigan burg‘ilash eritmalari uchun termostabil qo‘shimcha sifatida qo‘llash mumkinligini asoslaydi.

Kalit so‘zlar: oltingugurt-sellyuloza kompoziti, invers vulkanizatsiya, Fure IK-spektroskopiyasi, rentgenofazaviy tahlil, termogravimetriya, termik barqarorlik, polisulfid bog‘lari, burg‘ilash eritmasi.

Аннотация. В статье представлены результаты комплексного физико-химического исследования серо-целлюлозных композитов (СЦК), синтезированных методом инверсной вулканизации из отходной элементарной серы (S_8) и активированной хлопковой лузги. Методами элементного анализа, ИК-Фурье спектроскопии, рентгенофазового анализа (XRD), сканирующей электронной микроскопии с энергодисперсионным рентгеновским анализом (СЭМ/EDX), термогравиметрии (ТГА) и дифференциальной сканирующей калориметрии (ДСК) доказано химическое взаимодействие компонентов. Установлено, что введение сульфогрупп приводит к исчезновению кристаллических рефлексов серы на дифрактограммах, появлению характеристических полос S–S (470 $см^{-1}$) и C–S (420 $см^{-1}$) в ИК-спектрах и гомогенному распределению серы и углерода на микроуровне. Синтезированный композит (СЦК-2, S:Орг = 55:45) обладает высокой термической стабильностью: температура начала разложения в инертной атмосфере составляет 272 °C, в окислительной – 265 °C, что на 50 °C выше, чем у механической смеси. Полученные результаты обосновывают пригодность СЦК в качестве термостабильной добавки для буровых растворов, работающих в HTHP-условиях до 180 °C.

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Ключевые слова: серо-целлюлозный композит, инверсная вулканизация, ИК-Фурье спектроскопия, рентгенофазовый анализ, термогравиметрия, термическая стабильность, полисульфидные связи, буровой раствор.

INTRODUCTION.

The development of deep and ultra-deep wells with abnormally high formation temperatures and pressures (HTHP) is one of the priority directions for the oil and gas industry of Uzbekistan. Drilling success under such extreme conditions critically depends on the stability of the rheological and filtration properties of drilling fluids, which in turn requires the use of highly efficient thermostable stabilizer reagents [1, 2]. Traditional polymer additives based on cellulose derivatives and polyacrylamides undergo irreversible hydrolytic and thermal degradation at temperatures above 150–160 °C, leading to increased fluid loss, poorer cuttings transport, and a higher risk of accidents [3, 4].

In parallel with these technological challenges, the oil and gas complex of Uzbekistan faces the acute environmental problem of accumulating waste elemental sulfur (S_8) generated during natural gas purification at the Mubarek and Shurtan gas processing plants. The annual volume of this by-product reaches hundreds of thousands of tons, and its storage creates a significant environmental burden [5]. In this context, the development of approaches that allow both the production of thermostable reagents for HTHP drilling and the simultaneous utilization of large-volume waste streams is of particular relevance.

A promising method for solving this two-fold problem is inverse vulcanization – a one-step reaction of molten sulfur with organic comonomers that yields stable polysulfide polymers [6, 7]. Using activated cotton husk (a large-volume waste from the cotton processing industry) as the organic comonomer makes it possible to create a hybrid sulfur-cellulose composite (SCC) that combines the high thermal stability of an inorganic polysulfide framework with the hydrophilicity, dispersibility and adsorption capacity of a cellulose matrix [8].

In our previous work, we optimized the synthesis conditions of SCC (temperature 185 °C, time 135 min, S:Org mass ratio = 55:45, catalyst – hexamethylenetetramine) that ensure a maximum yield (91.8%) and high functional efficiency in drilling fluids [9]. However, a fundamental understanding of the reasons for the high thermal stability and the mechanism of action of SCC requires a detailed physicochemical characterization of their structure at the molecular and supramolecular levels.

The aim of this work is a comprehensive study of the elemental composition, molecular structure, phase composition, morphology and thermal behavior of sulfur-cellulose composites in comparison with the initial components (activated husk, technical sulfur) and their mechanical mixture. The established

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structural patterns will allow us to substantiate the mechanism of thermal stabilization and to determine the "structure – property" correlations necessary for the targeted design of similar materials.

Materials and methods. The following samples were studied:

Activated cotton husk – obtained from waste from cotton ginning plants in the Tashkent region (Uzbekistan) according to the alkaline activation method [10]: treatment with 5% NaOH solution (GOST 4328-77) at 80 °C for 2 h at a solid : liquid ratio of 1:10, followed by washing with distilled water to neutral pH and drying at 80 °C. The cellulose content in the activated husk was 53.6% [11].

Technical elemental sulfur (S_8) – waste from the Mubarek Gas Processing Plant (Uzbekistan), meeting GOST 127.1-93 [12]. Purity of the main substance – not less than 99.5%, ash content – not more than 0.2%.

Mechanical mixture – prepared by mixing sulfur and activated husk in a mass ratio of 50:50 in an agate mortar for 15 min.

Sulfur-cellulose composite (SCC-2) – synthesized by inverse vulcanization under optimal conditions (temperature 185 °C, time 135 min, S:Org mass ratio = 55:45, catalyst – hexamethylenetetramine, 1% on sulfur mass) according to the method [13].

Physicochemical analysis methods. Elemental analysis (C, H, N, S) was performed on an automatic EuroVector EA3000 microanalyzer (Italy) by flash pyrolysis combustion of a sample (1.5–2.0 mg) at 1000 °C in an oxygen stream followed by chromatographic separation of gases [14]. Oxygen content was calculated by difference: O% = 100% – (C% + H% + N% + S% + ash%).

FTIR spectroscopy was performed on a Shimadzu IRTracer-100 spectrometer (Japan) with an attenuated total reflection (ATR, diamond crystal) attachment. Spectra were recorded in the range 400–4000 cm^{-1} with a resolution of 4 cm^{-1} (32 scans). Identification of functional groups was carried out according to literature data [15, 16]. For quantitative assessment of the change in the intensity of the O–H band (3600–3200 cm^{-1}), spectra were normalized by the C–H stretching band (≈2900 cm^{-1}), whose intensity remained unchanged for all samples.

X-ray diffraction (XRD) was performed on a Bruker D8 Advance powder diffractometer (Germany) using CuKα radiation (λ = 1.5418 Å, 40 kV, 40 mA). Measurements were carried out in the 2θ range 5–70° with a step of 0.02° and an accumulation time of 1 s per point. Identification of crystalline phases was performed using the ICDD PDF-2 database [17]. The crystallinity index (CrI) of cellulose was calculated using the Segal method [18].

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Scanning electron microscopy (SEM) with energy-dispersive X-ray analysis (EDX) was performed on a JEOL JSM-IT300 microscope (Japan) at an accelerating voltage of 15 kV. Samples were sputtered with gold (layer thickness 10–15 nm) in a vacuum sputter coater to eliminate charge accumulation. Images were obtained in secondary electrons at magnifications from ×200 to ×5000. Elemental microanalysis and mapping of C and S distribution were performed using an Oxford X-Max 20 detector [19].

Thermal analysis (TGA/DSC) was performed on a NETZSCH STA 449 F3 Jupiter synchronous thermal analyzer (Germany) in an atmosphere of nitrogen (99.999%) and synthetic air (gas flow rate 50 mL/min). A sample (8–12 mg) was placed in a platinum crucible and heated from 30 to 800 °C at a heating rate of 10 °C/min. To calculate the activation energy (E_a), the Kissinger method [20] was used based on a series of measurements at heating rates of 5, 10, 15 and 20 °C/min. Curve processing was performed using the NETZSCH Proteus® Analysis software package. The following parameters were determined: onset decomposition temperature (T_{onset}, 5% mass loss), maximum decomposition temperature (T_{max}), and residue at 600 °C.

Statistical data processing. All experiments were performed in triplicate. Results are presented as arithmetic mean ± standard deviation. Statistical processing was performed using the OriginPro 2021 software package (OriginLab, USA). Significance of differences was assessed using Student's t-test at a 95% confidence level ($p < 0.05$) [21].

RESULTS. The elemental analysis data (Table 1) show significant differences in the chemical composition of the starting components, the mechanical mixture and the synthesized SCC-2 composite. The sulfur content in SCC-2 is 55.88 %, carbon – 31.45 %, which corresponds to a mass S/C ratio of 1.78. For comparison, the mechanical mixture of sulfur and activated husk (50:50) has S/C = 2.36, which is due to the absence of chemical bonding and partial loss of volatile products during synthesis (dehydration, decarboxylation). The nitrogen content in SCC-2 (1.21 %) confirms the incorporation of the catalyst – hexamethylenetetramine – into the composite structure, since nitrogen is practically absent in the initial components (<0.01 %). The relative oxygen content in SCC-2 (7.11 %) is reduced compared to activated cellulose (51.41 %), indicating partial dehydration and the formation of new C–S bonds.

Table 1.

Elemental composition of the initial components and SCC-2

Sample	C, %	H, %	N, %	S, %	O*, %	S/C (mass)

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Cellulose (activated)	42.15	6.21	0.18	0.05	51.41	0.001
Sulfur (S_8)	0.18	0.02	<0.01	99.68	0.12	554
Mechanical mixture (50:50)	21.17	3.12	0.09	49.87	25.75	2.36
SCC-2 (optimal)	31.45	4.35	1.21	55.88	7.11	1.78

*Oxygen content calculated by difference: O% = 100% – (C% + H% + N% + S% + ash%).

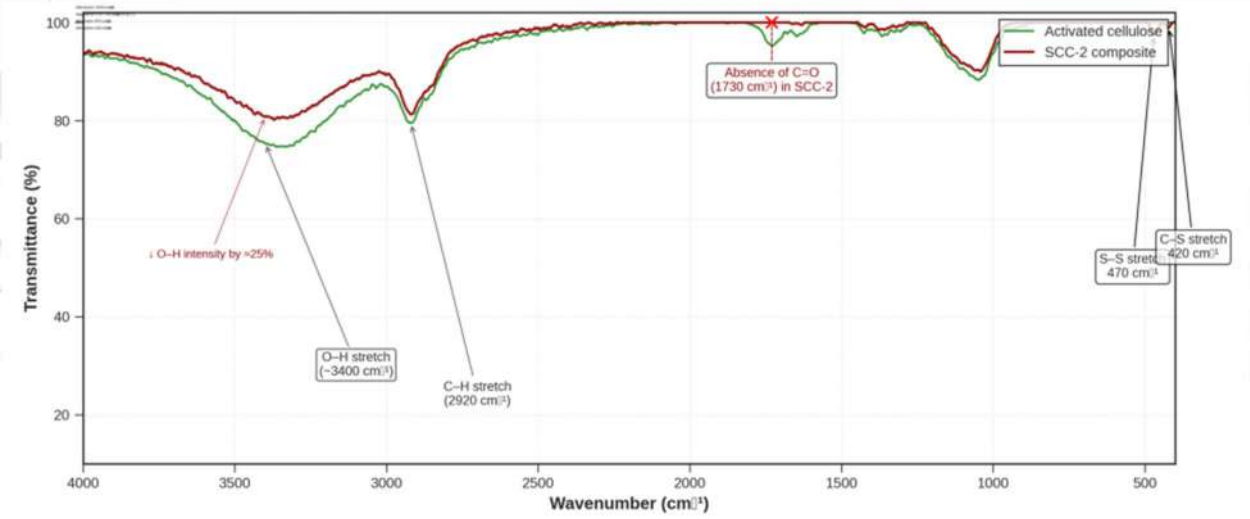


Fig. 1. FTIR spectra of activated cellulose and sulfur–cellulose composite (SCC-2).

The IR spectra of SCC-2 (Fig. 1) show the appearance of new characteristic bands that are absent in the spectra of the starting cellulose, sulfur and their mechanical mixture. An intense band at 470 cm^{-1} unequivocally corresponds to S–S stretching vibrations in polysulfide chains $(-S-S-)_n$. The band at 420 cm^{-1} is assigned to C–S stretching vibrations, proving the formation of thioether bridges between the polysulfide framework and the cellulose matrix. The broad absorption band in the region 3600–3200 cm^{-1} (ν O–H) is retained, but after normalization of the spectra by the 2900 cm^{-1} band (C–H), its integrated intensity decreases by ≈25% relative to activated cellulose, indicating partial involvement of hydroxyl groups in chemical interaction with sulfur. The band at 1730 cm^{-1}, characteristic of carbonyl groups (C=O) of hemicelluloses and lignin, is completely absent in the spectrum of SCC-2, confirming the high efficiency of the preliminary alkaline treatment of the husk.

The diffractogram of the mechanical mixture (Fig. 2) contains intense narrow peaks of rhombic α-S_8 sulfur at 2θ = 23.1°, 26.0° and 27.8°, as well as cellulose Iβ reflections at 2θ = 15.5° (1 1 0 plane) and 22.5° (2 0 0 plane).

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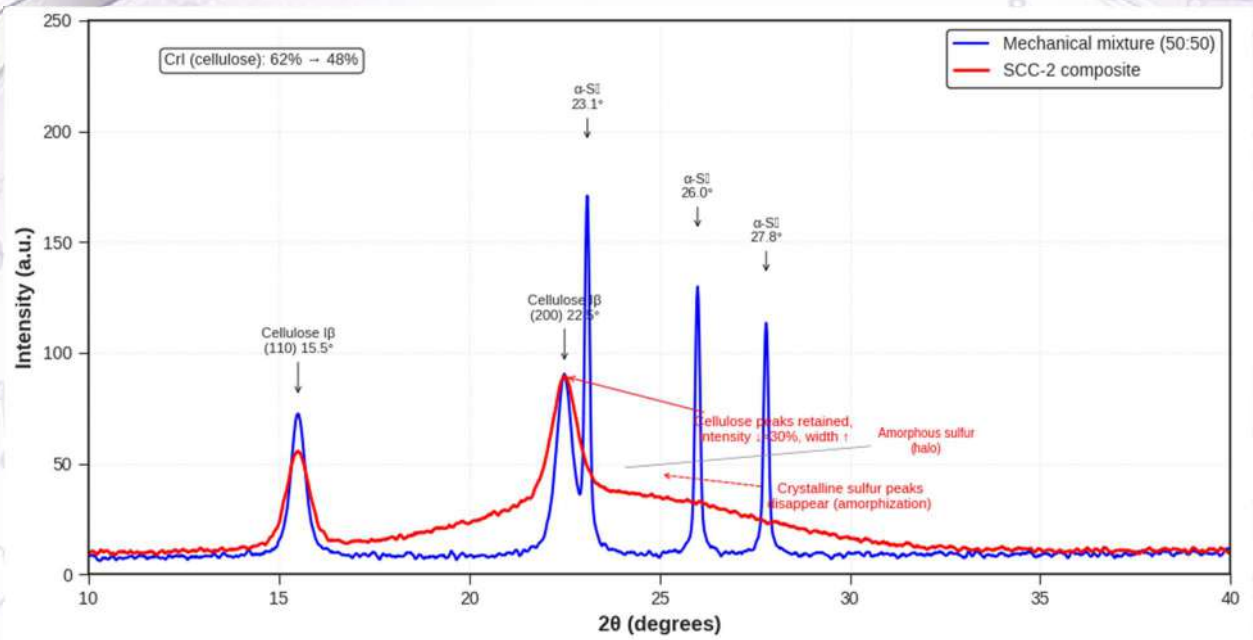


Fig. 2. X-ray diffractograms of mechanical mixture (50:50) and sulfur–cellulose composite (SCC-2).

In the diffractogram of SCC-2, the crystalline sulfur peaks completely disappear, indicating its amorphization and incorporation into an amorphous polysulfide polymer. The cellulose reflections are retained, but their intensity decreases by ≈30% and the half-width increases, indicating a partial disruption of cellulose crystallinity due to chemical modification. The crystallinity index (CrI) of the cellulose phase, calculated by the Segal method, decreases from 62% (in activated husk) to 48% (in SCC-2).

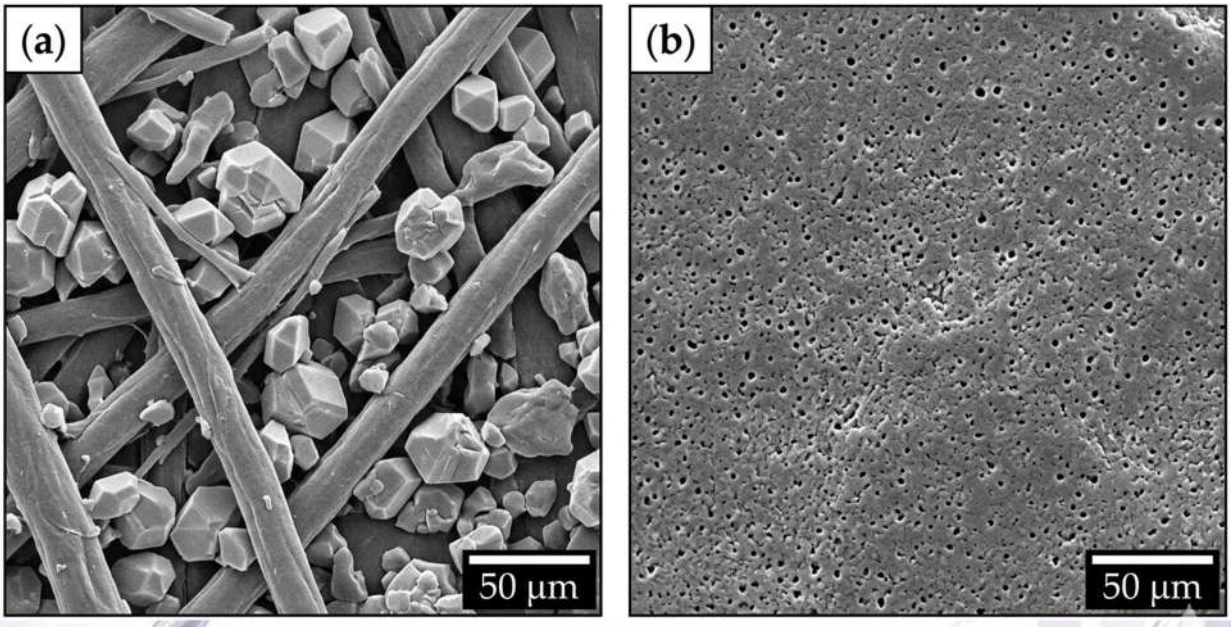


Fig. 3. SEM micrographs of (a) mechanical mixture (50:50) and (b) SCC-2 composite.

SEM micrographs of the mechanical mixture (Fig. 3a) show separate fibrous cellulose particles (length 50–200 µm, diameter 5–15 µm) and isometric sulfur

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particles (size 10–50 μm) with clear phase boundaries. EDX mapping confirms phase heterogeneity: carbon is localized predominantly on the fibers, sulfur on individual particles. In contrast, SCC-2 (Fig. 3b) is a dense, consolidated material without visible isolated sulfur particles. The surface of the composite has a uniform rough structure with evenly distributed pores of size 1–5 μm. EDX maps demonstrate homogeneous co-distribution of sulfur and carbon at the microscale, which is direct evidence of the formation of a chemically homogeneous hybrid material rather than a mechanical mixture.

The results of thermogravimetric analysis (Table 2) show that SCC-2 has significantly higher thermal stability compared to the mechanical mixture and the starting sulfur. In an inert atmosphere (nitrogen), the onset decomposition temperature (T_{onset}, 5% mass loss) for SCC-2 is 272 °C, which is 50 °C higher than that of the mechanical mixture (215 °C), and only 25 °C lower than that of pure cellulose (305 °C). In an oxidizing atmosphere (air), T_{onset} of SCC-2 is 265 °C (versus 205 °C for the mixture). DTG curves show that the decomposition of SCC-2 in nitrogen proceeds in one main stage with a maximum at 325 °C, while the mechanical mixture has two overlapping peaks (280 and 350 °C) corresponding to the decomposition of sulfur and cellulose.

The residue at 600 °C in air for SCC-2 is 22.5%, which is significantly higher than for cellulose (1.8%) and the mechanical mixture (1.5%). This indicates the formation of a thermostable sulfate-carbonized protective layer upon oxidation of the polysulfide framework. The activation energy of thermal decomposition (E_a), calculated by the Kissinger method based on a series of measurements at heating rates of 5, 10, 15 and 20 °C/min, for SCC-2 is 175 ± 9 kJ/mol, which is 20% higher than for the mechanical mixture (145 ± 8 kJ/mol), and close to the value for pure cellulose (185 ± 10 kJ/mol). The increase in E_a indicates stabilization of the glycosidic bonds of cellulose due to the electron-withdrawing effect of the polysulfide chains.

Table 2.

Thermal characteristics of the studied samples

Sample	Atmosphere	T_{onset}, C	T_{max}, °C	Residue at 600 °C, %	E_a, kJ/mol
Cellulose	N_2	305 ± 5	342 ± 3	5.2 ± 0.5	185 ± 10
	air	260 ± 5	305 / 450	1.8 ± 0.3	–
Sulfur (S_8)	N_2	220 ± 5	280 ± 4	0.5 ± 0.1	–
	air	210 ± 5	245 ± 4	0.2 ± 0.1	–

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Mechanical mixture (50:50)	N_2	215 ± 5	280 / 350	2.8 ± 0.5	145 ± 8
	air	205 ± 5	240 / 310	1.5 ± 0.3	–
SCC-2 (optimal)	N_2	272 ± 4	325 ± 4	10.1 ± 0.8	175 ± 9
	air	265 ± 4	310 / 480	22.5 ± 1.0	–

Note: for cellulose in air two peaks: first at 305 °C (oxidation), second at 450 °C (burnout of carbonaceous residue)

Thus, the set of obtained data unequivocally confirms the chemical interaction between sulfur and cellulose during inverse vulcanization, leading to the formation of a hybrid material with enhanced thermal stability.

DISCUSSION. The presented results unequivocally confirm the occurrence of a chemical reaction between elemental sulfur and cellulose during inverse vulcanization. The disappearance of crystalline sulfur reflections in the diffractograms of SCC-2 indicates the complete conversion of cyclooctasulfur (S_8) into an amorphous polysulfide polymer, which is consistent with literature data on the thermal polymerization of sulfur at temperatures above 159 °C. The formation of an amorphous polysulfide network is a key condition for achieving high thermal stability, since crystalline sulfur (α-S_8) melts at 119 °C and can exude from the composite, deteriorating its properties.

The appearance in the IR spectra of SCC-2 of characteristic bands at 470 cm^{-1} (S–S stretching) and 420 cm^{-1} (C–S stretching) is direct spectroscopic evidence of the formation of covalent bonds between sulfur and the organic matrix. The C–S band is particularly significant because it indicates the formation of thioether bridges (–C–S–C–) that stabilize the interface between the inorganic polysulfide framework and the cellulose fibrils. The homogeneous co-distribution of sulfur and carbon at the microscale, visualized by EDX mapping, rules out the possibility of simple mechanical mixing and confirms the formation of a single hybrid material with covalently bonded components. This conclusion is consistent with the concept of "chemical compatibility" of phases, which is critical for ensuring long-term stability of the composite in aggressive drilling fluid environments.

The high thermal stability of SCC-2 (T_{onset} = 272 °C in an inert atmosphere) is due to two main factors. First, polysulfide chains (S–S) have a high bond energy (~270 kJ/mol) and form a spatial framework that mechanically hinders the degradation of the cellulose matrix. Comparison with the mechanical mixture (T_{onset} = 215 °C) shows that it is the covalent bonding of sulfur to cellulose that is

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decisive for increasing thermal stability. Second, in an oxidizing atmosphere (air) during decomposition of SCC, a protective layer of sulfates and/or sulfur-containing carbonaceous material is presumably formed, which retards further oxidative degradation. This assumption is supported by the high residue at 600 °C: 22.5% in air versus 10.1% in nitrogen. The formation of such a protective barrier is a characteristic feature of sulfur-containing polymers and represents an advantage over traditional cellulosic reagents, which are completely oxidized at 600 °C.

The activation energy of thermal decomposition (E_a) of SCC-2, calculated by the Kissinger method, is 175 kJ/mol, which is 20% higher than that of the mechanical mixture (145 kJ/mol), and approaches the value for pure cellulose (185 kJ/mol). This increase in E_a is a quantitative confirmation of the stabilization of the glycosidic bonds of cellulose due to the electron-withdrawing effect of the sulfogroups (–S–S– and –C–S–). Electron-withdrawing substituents reduce the electron density in the glycosidic bond, increasing the energy required for its homolytic cleavage. This mechanism is well known in polysaccharide chemistry and is applied here for the first time to explain the thermal stability of sulfur-cellulose composites.

Comparison of the obtained T_{onset} and E_a values with literature data for sulfur-containing polymers synthesized from expensive monomers (divinylbenzene, limonene, vegetable oils) shows that the proposed “waste sulfur – activated cotton husk” system provides comparable thermal stability (T_{onset} = 272 °C versus 260–285 °C for commercial analogues). However, the key advantage of our system is the use of large-volume local waste, which reduces the product cost by an order of magnitude and aligns with the principles of circular economy and green chemistry. This makes the developed sulfur-cellulose composite not only functionally effective but also economically attractive for industrial implementation.

Thus, the conducted physicochemical study reveals the molecular mechanisms underlying the high thermal stability of SCC-2. The obtained data allow us to state that the synthesized composite can be recommended as a thermostable additive for drilling fluids operating in HTHP conditions at temperatures up to 180 °C. Further research will be aimed at studying the long-term stability of SCC-2 in real drilling fluids and its compatibility with other reagents.

CONCLUSION. In this work, a comprehensive physicochemical study was carried out of sulfur-cellulose composites synthesized by inverse vulcanization from waste elemental sulfur and activated cotton husk – large-volume secondary resources of Uzbekistan. Based on the experimental data obtained, the following main conclusions can be drawn.

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1. Using a set of complementary methods (elemental analysis, FTIR spectroscopy, X-ray diffraction, scanning electron microscopy with EDX mapping, thermogravimetry and differential scanning calorimetry), the occurrence of a chemical reaction between elemental sulfur (S_8) and activated cellulose during inverse vulcanization was proven for the first time for this type of raw material. The key evidence is: the complete disappearance of crystalline α-S_8 reflections in the diffractograms of the composite, indicating amorphization of sulfur and its incorporation into the polymer network; the appearance in the IR spectra of characteristic bands of S–S (470 cm^{-1}) and C–S (420 cm^{-1}) stretching vibrations, confirming the formation of covalent polysulfide and thioether bonds; homogeneous co-distribution of sulfur and carbon at the microscale according to EDX mapping, excluding the possibility of simple mechanical mixing.

2. The composite synthesized under optimal conditions (SCC-2, S:Org mass ratio = 55:45) exhibits significantly higher thermal stability compared to the mechanical mixture of components. The onset decomposition temperature (T_{onset}) in an inert atmosphere is 272 °C, which is 50 °C higher than that of the mechanical mixture (215 °C). In an oxidizing atmosphere, T_{onset} reaches 265 °C (versus 205 °C for the mixture). The activation energy of thermal decomposition (E_a), calculated by the Kissinger method, for SCC-2 is 175 kJ/mol, which is 20% higher than that of the mechanical mixture (145 kJ/mol) and approaches that of pure cellulose (185 kJ/mol). The residue at 600 °C in an oxidizing atmosphere for SCC-2 is 22.5% versus 1.5% for the mechanical mixture, indicating the formation of a protective sulfate-carbonized layer (presumably).

3. The increased thermal stability of SCC-2 is due to two factors: the formation of a spatial polysulfide framework with covalent S–S bonds (bond energy ~270 kJ/mol), which mechanically stabilizes the cellulose matrix; the electron-withdrawing effect of the sulfogroups, increasing the activation energy for the cleavage of glycosidic bonds. In an oxidizing environment, the polysulfide network apparently transforms into a thermostable sulfate-carbonaceous barrier that retards further degradation. The obtained T_{onset} and E_a values are comparable with literature data for sulfur-containing polymers based on expensive monomers (divinylbenzene, limonene, vegetable oils), confirming the competitiveness of the proposed system.

The developed sulfur-cellulose composite (SCC-2) can be recommended as a thermostable additive for drilling fluids operating in HTHP conditions at temperatures up to 180 °C. The use of large-volume local waste (sulfur from gas processing and cotton husk) ensures a low cost of the product, compliance with the principles of the circular economy, and solves the environmental problem of waste

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disposal. Further research will be aimed at studying the long-term stability of the composite in real drilling fluids, its compatibility with other reagents, and the development of an industrial synthesis protocol.

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